IMPACTS OF SOIL PH ON WINTER CANOLA CULTIVARS IN THE SOUTHERN GREAT PLAINS

By

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Abstract: Winter canola (*Brassica napus* L.) is an important rotational crop for wheat systems in the Southern Great Plains, which possess a wide range of acidic soils. However, unlike many other crops, most winter canola cultivars have not been evaluated for pH and Al^{3+} tolerance. Four field trials were established over two growing seasons to evaluate four winter canola cultivars on pre-adjusted pH gradients. Generally, canola yields were positively influenced by increasing soil pH, with high yields coming from more neutral pH. However, response of canola yields to pH differed by location and cultivar. Critical soil pH was only found at one site year, with critical pH values much lower than previous evaluations (pH 3.90). Environmental conditions at planting paired with fewer data points above could have contributed to this variable response. At the Chickasha location, soil pH and extractable Al had a limited relationship, with low levels of Al^{3+} found, indifferent of soil pH (r^2= 0.03). Variation in yields at Chickasha were potentially better explained by other production factors, not changing pH. In addition, a greenhouse study was established to analyze the potential of poultry litter biochar to alleviate extractable Al^{3+} in soil systems. Four biochar treatments were analyzed: 2.24 Mg ha^{-1}, 5.6 Mg ha^{-1}, and 11.21 Mg ha^{-1} as well as a control. In the greenhouse evaluation, 2.24 Mg ha^{-1} biochar application had a positive effect on alleviating extractable Al^{3+} from the soil. Higher application rates of biochar on soil also alleviated Al^{3+} concentration, but not at a significant amount to justify the necessity of higher application rates. Applications of 2.24 Mg ha^{-1} reduced Al^{3+} concentration by 28.23 mg kg^{-1} while at an application of 5.6 Mg ha^{-1} concentrations were only further reduced by 10.07 mg kg^{-1}. Results from these studies indicated canola grain yields were impacted by the factors associated with soil acidity. These results highlight the continual evaluation of currently and newly available winter canola cultivars for their tolerance of soil acidity. Furthermore, growers should be knowledgeable of canola cultivar, soil pH and exchangeable Al^{3+} when determining the feasibility of winter canola in their production systems.

TABLE OF CONTENTS

Chapter Page

I. INTRODUCTION 1

II. REVIEW OF LITERATURE 3

Soil pH and Classification 3
Formation of Acidic Soils 4
Impacts of Soil Acidity on Crops 7
Effects of Soil pH on Winter Canola 8
Oklahoma Soils 9
Correcting Acidic Soils 11
Alternative Liming Sources 13
Biochar 13

III. METHODOLOGY 15

Field Study 15
Field Study Soil Analysis 17
Field Study Statistical Analysis 18
Greenhouse Study 18
Greenhouse Soil Analysis 19
Greenhouse Statistical Analysis 19

IV. RESULTS AND DISCUSSION 20

Weather 20
Crop Yield 23
Soil pH and Yield 23
Impact of Soil pH on Individual Cultivars 25
Soil pH and Al 28
Al and Crop Yield 29
Greenhouse Results 30

V. CONCLUSION 33

Field Study 33
Greenhouse Study 34

Chapter Page

REFERENCES 36

APPENDICES 41

LIST OF TABLES

Table Page

Common descriptive classes of soil pH and their ranges 4
Different liming materials, their chemical composition, and percent calcium carbonate equivalent 12
The approximate amount of finely ground lime needed to raise the pH of 7-inch layer of soil 13
Coordinates, soil series, and soil taxonomic classes for trial locations 16
pH gradients for each location after adjustment and trials 16
Relationships between soil pH and yield, averaged across locations 25
Dekalb winter canola cultivars analyzed, listed with their corresponding color for identification in following figures and tables 25
Relationships between soil pH and yield across all locations in 2015-16 and 2016-17 season....26
Analysis of poultry litter biochar used in greenhouse trial 31

LIST OF FIGURES

Figure Page

Average soil pH for each county in Oklahoma....10
Average soil pH for Oklahoma....11
Rainfall and Temperature for Stillwater (EFAW)....21
Rainfall and Temperature for Stillwater (N40)....22
Rainfall and Temperature for Perkins....22
Rainfall and Temperature for Chickasha....23
Relationship between soil pH and winter canola yield....24
Relationships between pH and yield for each cultivar at Perkins....27
Relationship between soil pH and extractable Al^{3+} at each location....28
Al and crop yield for each location....30
Biochar replication averages with statistical significances....32

CHAPTER I

INTRODUCTION

Hard red winter wheat (*Triticum aestivum* L.) is the primary crop grown in the United States Great Plains, with over 12 million hectares being grown across the region in 2017 (NASS, 2017). Oklahoma is one of the primary wheat producing states, planting over 2 million hectares (NASS, 2017). While wheat will continue to be the dominant crop throughout the region, overall productivity has seen decreased seed quality, increased pest pressure, and a stagnation in yields (Patrignani et al., 2014). These issues are attributed to the lack of cropping diversity within these production systems, as wheat has been primarily grown in monoculture systems for decades. During this period, several rotational crops have been introduced to the region in an attempt to increase the diversity of the cropping systems. However, many of these other crops have not been successful due to challenging environmental conditions, particularly when integrating summer crops in these rotations.

Winter canola (*Brassica napus* L.) was introduced to the region in the early 2000s as a potential rotational crop for winter wheat and has been able to overcome many of the challenges that other rotation crops have faced. Not only is canola a broadleaf, providing a break in weed,

insect, and disease cycles, but canola also adds the advantages of having similar management and production practices to winter wheat (Edwards et al., 2006; Liebman and Dyck, 1993).

While the physiology of the crop and management similarities show promise in canola, several challenges exist that have limited widespread adoption of the crop within the region. These challenges are focused around the instability of canola yield in arid and semi-arid regions. The lack of yield stability has been associated with the increased sensitivity of winter canola to abiotic stresses. One of these stresses is soil pH. Winter wheat has the ability to sustain growth and yields at soil pH of 5.5 or lower (Zhang and Raun, 2006; Lollato et al., 2013). Initial evaluations of winter canola in Oklahoma documented that the critical soil pH levels were higher as well as had a lower critical soil Al^{3+} concentration compared to winter wheat (Lofton et al., 2008). The challenges of winter canola compared to winter wheat increase as several winter wheat varieties have been identified as low pH tolerant. Integrating these less sensitive wheat varieties into production systems dissuades the application of lime to correct for a problematic pH, as variety selection is typically a cheaper option. As opposed to wheat, breeding for acid tolerance has not been a primary focus for winter canola breeding programs. This further increases the difficulty of integrating canola into traditional wheat production systems in Oklahoma and the southern Great Plains.

Understanding the impacts that soil pH and available Al^{3+} have on winter canola productivity will continue to be a critical aspect needing to be addressed for increased adoption and management as a rotational crop for wheat production systems. Furthermore, documenting the potential response of common commercially available winter canola cultivars to soil pH and Al^{3+} will prove to be a valuable tool for managing soil pH in winter canola systems. Therefore, the objectives of this study were to 1) Document the impact of soil pH and Al^{3+} on winter canola productivity and yield, 2) Evaluate the response of four winter canola cultivars to varying soil pH, and 3) Determine the ability of biochar to alleviate extractable Al^{3+} concentrations in soils.

CHAPTER II

LITERATURE REVIEW

Soil pH and Classification

Many soil scientists consider soil pH the most influential soil factor, with it frequently called the master soil variable (Brady and Weil, 2006). Soil pH is a scale that is the indirect measure of H^+ concentration in the soil solution. The more H^+ ions present, the more acidic the soil, while the lower the number of H^+ ions, the more basic the soil. Soil pH is typically demonstrated on a logarithmic scale and determined by calculating the $-\log[H^+]$.

$$pH = \log\frac{1}{[H^+]} = -\log[H^+]$$

Therefore, a solution with $H^+ = 10^{-6}$ M has a pH of 6.0.

$$10^{-6}\ M \rightarrow -\log[10^{-6}] = -[-6] = 6.0$$

(Modified from Havlin et al., 2015)

A soil pH value of 7.0 is considered neutral while solutions with values <7.0 are acidic and those >7.0 are basic or alkaline. Other nomenclature for soil pH is available that describes the amount of H^+ in the soil and how the soil pH level will alter biologic growth. Table 1 gives common descriptive classes of soil pH.

Table 1 Common descriptive classes of soil pH and their ranges (Modified from Havlin et al., 2015).

Descriptive Term	Soil pH range
Extremely acidic	< 4.5
Very strongly acidic	4.6-5.0
Strongly acidic	5.1-5.5
Moderately acidic	5.6-6.0
Slightly acidic to neutral	6.1-7.3
Slightly alkaline	7.4-7.8

Soil pH can be determined by diluting soil in deionized water or a dilute salt solution, usually $CaCl_2$. This can be done in either a 1:1 or 1:2solution ratio. Soil pH measured using $CaCl_2$ is usually lower, but the salt solution displaces additional H^+ from exchange sites, but also can be more stable due to being less susceptible to moisture and salt content variations.

Formation of Acidic Soils

Soil acidity is classified into three "groups" of acidity, classified as active, exchangeable, and residual acidity (Brady and Weil, 2002). Active acidity is the H^+ and Al^{3+} in the soil solution and actively interacts with plants and is involved in chemical reactions. Exchangeable acidity is the H+ and Al^{3+} that is found on the exchange sites in relation with soil particles. This group of soil acidity is not actively involved in chemical and biological processes in the soil system but can quickly be transferred into active acidity through exchange with other cations. Residual acidity is often the largest fraction of soil acidity within the soil system as well as has the slowest availability to interact within the soil system. This form of acidity is categorized as the non-exchangeable Al^{3+} that is bound in the crystalline structure of clays and organic matter (Havlin et al., 2005). When evaluating and correcting soil acidity at a minimum active and exchangeable acidity must be evaluated. This is because the soil acts as a buffer against soil pH change. When a neutralization agent is added to the soil, H^+ molecules from the exchange sites are released, acidifying the soil system; therefore, buffering the soil against the pH change. The amount of

buffering and soil neutralization is highly dependent on how much pH is within the exchangeable category.

The formation of acidic soils is largely due to soil additions and subtractions such as ammonia/ammonium fertilizers and nutrient uptake by crops, product removal during most agricultural processes, soil organic matter (OM), and leaching and precipitation (Miller et al., 2009 and Havlin et al., 2015). The formation of acidic soils can also stem from many other factors pertaining to soil parent material or increase their potential to develop acidity naturally due to their inherently low cation exchange (Sumner and Noble, 2003).

Nutrient uptake by crops and the addition of fertilizers are both factors that can lead to soil acidity in production systems. Addition of ammonia/ammonium N fertilizers in high amounts is a large factor in the acidification of many soils in more intense production areas (Brown et al., 2008). As nitrification occurs, N from ammonia (NH_3) and ammonium (NH_4^+) fertilizers is converted into nitrates (NO_3^-) and H^+ ions are released into the soil, resulting in acidification (Brady and Weil, 2008). This can be altered based on the cation exchange capacity (CEC) of a given soil. The CEC determines how many exchangeable cations the soil can absorb (Brady and Weil, 2008). As plants take up cations from the soil, they release protons (H^+) to maintain electrical neutrality, while when plants uptake anions such as nitrates, hydroxyls (OH^-) are released and neutralize any free protons (Miller et al., 2009). However, plants do not have the capacity to uptake all of the anions, so protons are allowed to collect and build, causing soil acidification due to high N inputs (Miller et al., 2009). When a plant uptakes more cations (K^+, NH_4^+, Ca^{2+}, etc.) than anions, an excess of H^+ ions are released into the rhizosphere, causing the soil to be more acidic; while when a plant uptakes more anions (NO_3^-, SO_4^-, among others) than cations, an excess of OH^- ions are released, causing a more basic or alkaline soil (Havlin et al., 2013 and Brady and Weil, 2008). In a lower CEC soil, the anion exchange capacity is higher, resulting in a more acidic soil (Sparks, 2003).

Once a crop has taken up and exchanged nutrients from the soil, the biomass left in the field can also play a role in soil acidity. As crop reside is left in the field, microbial degradation takes place to break down the biomass, causing an increase of CO_2 in the soil which is tenfold the CO_2 in the atmosphere, reacting with water to create H^+ and HCO_3^-.

$$Organic\ C \rightarrow R-COOH \rightarrow R-COO^- + H^+$$

(Havlin et al., 2015).

The amount of acidity produced through organic matter breakdown is highly dependent on the type of organic matter. Organic matter under particular vegetation types, such as coniferous forest, have the potential to produce more acidic soil conditions compared to grasslands (Havlin et al., 2015). Some soil organic materials can contain carboxylic or phenolic compounds that can release H^+ into the soil solution, similar to that of weak acids (Havlin et al., 2015). Although most mineral soils, such as those in Oklahoma, contain a much lower amount of organic matter, thus this type of soil acidification is minimal compared to other fractions.

Rainfall and leaching play an important part in soil acidification as well. Acidification of the soil occurs when water containing nitrates (NO_3^-) are leached below the rooting zone (Havlin et al., 2015). Rainfall increases this process by adding more acidic components and providing more moisture for nitrate transport. Rainfall itself is naturally acidic because of the relationship between water and atmospheric CO_2, resulting in a pH of around 5.6; the H_2O/CO_2 relationship is as follows:

$$H_2O + CO_2 \leftrightarrow H_2CO_3 \leftrightarrow H^+ + HCO_3^-$$

(Havlin et al., 2015). However, since the added acid is weak and in very low quantities, there can be little to no response on bulk soil pH depending on the amount of rainfall received and the beginning pH of soil (Havlin et al., 2015).

Impacts of Soil Acidity on Crops

Soil pH can greatly influence plant growth and productivity alone; however, the greatest impact of soil pH is the influence it has on other soil chemistry components. Low soil pH causes an increase in availability and mobility of other micronutrients such as metals that are not readily available in a higher, above neutral soil pH. One of the more prominent of these is active Aluminum [Al^{3+}] concentration (Bolan and Hedley, 2003). This becomes problematic in most cropping systems due to the increasing concentration of Al^{3+} as soil pH decreases. When soil pH drops below 5.0, organic and inorganic Al^{3+} become more soluble in response to the lower pH, causing higher mobility and plant response as Al^{3+} toxicity (Li and Johnson, 2016; Sumner and Noble, 2003).

$$Al^{3+} + H_2O \leftrightarrow Al(OH)^{2+} + H^+$$

$$Al(OH)^{2+} + H_2O \leftrightarrow Al(OH)_2{}^{+} + H^+$$

$$Al(OH)_2{}^{+} + H_2O \leftrightarrow Al(OH)_3{}^{0} + H^+$$

$$Al(OH)_3{}^{0} + H_2O \leftrightarrow Al(OH)_4{}^{-} + H^+$$

(Havlin et al., 2015)

At each 1.0 decrease in soil pH, Al^{3+} concentration becomes 1000 times greater, also aiding in Al^{3+} toxicity (Raun and Zhang, 2006). Naturally high amounts of Al^{3+} in the soil system can result in significant declines in growth and nutrient uptake which can also lead to deficiencies (Zhang, 2017; Havlin et al., 2013). Due to the pH buffering processes in soil organic matter, systems with higher soil organic matter do not readily express signs of Al^{3+} toxicity compared to similarly acidic soils with lower soil organic matter (Godsey et al., 2007; Li and Johnson, 2016). The most common and easily identified symptom of Al^{3+} toxicity is root growth inhibition in the form of clubbed roots with little to no lateral growth, which reduces or restricts water and nutrient uptake by the plant (Sumner and Noble, 2003; Tang et al., 2007). Aluminum toxicity can also

block pathways for the plant to uptake Calcium [Ca^{2+}], a macronutrient that cannot be hindered due to its vitality to the plant's overall structure and effect on crop yield (Raun et al., 2000).

Low pH can also affect the availability of many other plant essential nutrients as well. At a lower pH, many nutrients are rendered unavailable to plants, being tied up by other elements in the soil, thereby increasing the availability of other nutrients in addition to the toxic elements (Plaster, 1997). For example, at a pH of 6.0, N, P, and K begin and continue to be exceedingly less available as pH decreases and elements like Al^{3+}, which is toxic to plants, become exceedingly more available (Plaster, 1997; Jones, 2012). Plant responses to low pH can vary based on individual crop. Hard red winter wheat is grown in moderately to strongly acidic soils in Oklahoma and sensitivities to soil acidity and exchangeable Al^{3+} concentration have been found to be cultivar specific (Lollato et al., 2013; Kariuki et al., 2007). However, winter canola is not as tolerant to low soil pH as winter wheat, indifferent of cultivar, and there is a breeding push for more acid-tolerant winter canola varieties to be able to withstand the same conditions as winter wheat.

Effects of Soil pH on Winter Canola

Winter canola cannot tolerate the same soil conditions as wheat, so even though it is a promising rotational crop from aspects of crop management, it can be difficult when considering crop management to individual soil properties. There have been very few efforts to determine how soil pH affects winter canola yield because soil acidity is not problematic in most traditional canola-producing areas and therefore is not a major concern in those breeding programs (Lofton et al., 2010). With continued adoption of winter canola within the region, several new cultivars have been established in the state that are drastically different from those documented by Lofton et al. (2008); however, they have not been evaluated for their pH sensitivities or tolerances. Currently, only conventional cultivars have been evaluated for their pH and Al^{3+} tolerances (Lofton et al., 2010). Lofton et al. (2010) documented pH sensitivities in conventional canola

cultivars, and found that canola yield began to decline at a pH of 5.8 with critical Al^{3+} concentration of 11.3 to 14.7 mg kg^{-1}.

Soil pH can affect many different plant growth processes as well as nutrient availabilities and disease presence and pressure pertaining to both winter and spring canola varieties. In addition to Al^{3+} toxicity, acidic soils can also cause Fe and Mn toxicities as well (Canola Council of Canada, 2017).

In addition to plant growth processes and nutrient availabilities, soil pH can affect disease presence and pressure as well as other properties. Perera et al. (2016) found that pH can greatly affect structural properties of oil proteins cruciferin and napin found in canola. Cruciferin and napin proteins are the most abundant proteins, contributing 20% (napin) and 60% (cruciferin) to the accumulated proteins in the plant (Hoglund et al., 1992; Perera et al., 2016). *Plasmodiophora brassicae* (*P. brassicae*) is a plasmodiophoromycete that is expressed as clubroot disease in cruciferous plants, including canola (Niwa et al., 2007). Symptoms of clubroot disease are visually similar to Al^{3+} toxicity in canola and can cause the similar yield decreases. Soils with a lower pH can cause spores to germinate at a quicker rate and infect more plants more rapidly (Rashid et al., 2013). Soil moisture and growing seasons with higher precipitation cause higher infection rates due to increasing the mobility of the spores in the soil solution (Dixon, 2009). Application of lime and other Ca-rich organic materials can aid in suppressing spore germination due to its ability to raise soil pH, which is the driving factor in *P. brassicae* spore germination suppression (Niwa et al., 2007).

Oklahoma Soils

Oklahoma producers are very commonly challenged with low pH soils across the state, but they are generally easier and relatively inexpensive to manage (Raun and Zhang, 2006). While a majority of the soils in the prominent agricultural production regions of Oklahoma are more acidic, this is generally due to intensive management and production and not the natural

state of the soil (Raun and Zhang, 2006). Acidic soils in Oklahoma are found more often across the central and western part of the state where most farming practices take place, while acidic soils on the east side of the state are more sporadic (Raun and Zhang, 2006). From 2000 to 2003, half of the reported soil pH in Oklahoma fell below 5.9 and half above, resulting in a median soil pH of 5.9, with 35% of samples having a soil pH lower than 5.5, which is a very low and critical level for most cropping systems (Raun and Zhang, 2006). Figure 1 illustrates data from Raun and Zhang, (2006).

More recent data has been published by Arnall and Phillips (2018), which indicated that the average soil pH across Oklahoma is 6.0-6.1 with up to 2.3 range in both directions. Figure 2 illustrates the most recent data from Arnall and Phillips, (2018). Because of the acidic nature of Oklahoma soils in many farming operations, soil amendments are needed but due to the high cost of transportation of agricultural lime, producers are not always able to amend their soil (Lollato et al., 2013).

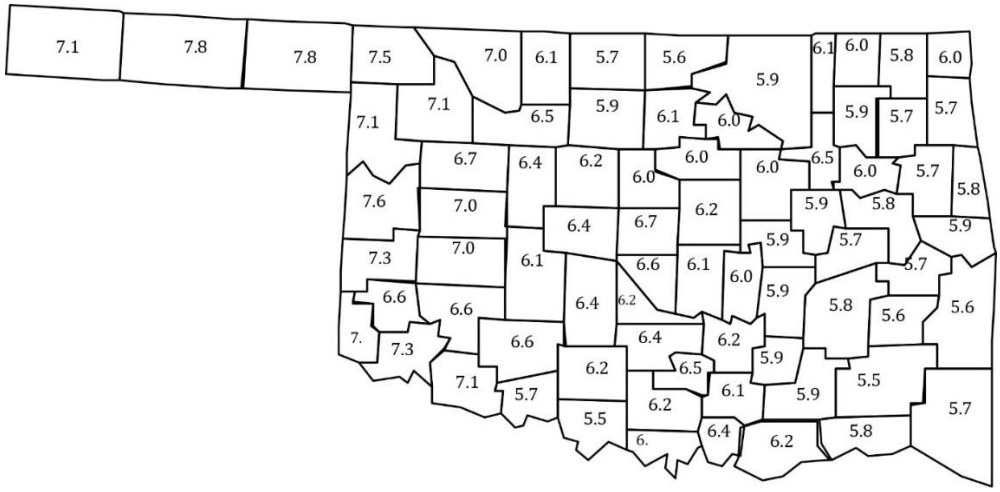

Figure 1 Median soil pH for each county in Oklahoma (Raun and Zhang, 2006; Used with Consent).

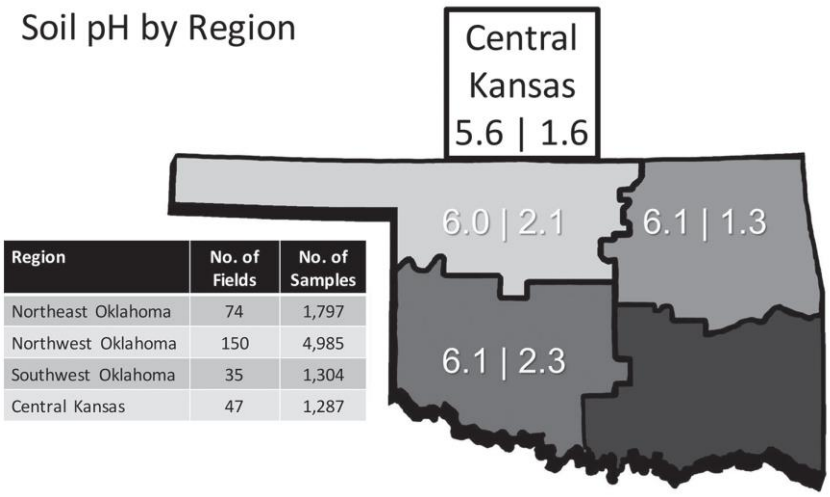

Figure 2 Average soil pH for Oklahoma. First value indicates average pH in its respective region. Second value indicates average pH range from aforementioned average pH per region (Arnall and Phillips, 2018; Used with Consent).

Correcting Acidic Soils

Liming and the addition of various soil amendments has been shown to alleviate issues with soil pH, including Al^{3+} toxicity (Tang et al., 2007). Several soil amendments have demonstrated the ability to aid in the neutralization of soil pH. Limestone or aglime is the most common liming agent utilized to neutralize acidic soils in agriculture production systems and has continuously been the most effective and most used due to lowest cost per ton of active ingredient (Lollato et al., 2013; Raun and Zhang, 2006). Most liming materials will contain oxides, hydroxides, or carbonates of base Ca or Mg forms of alkaline earth metals that form hydroxides in water such as MgO, CaO, $CaCO_3$, etc. (Brady and Weil, 2008). For example, in water, CaO reacts with water forming Ca^{2+} and releasing OH^- into soil solution, therefore raising soil pH:

$$CaO + H_2O \rightarrow Ca(OH)_2 \rightarrow Ca^{2+} + 2OH^-$$

(Brady and Weil, 2008)

The active ingredient in liming materials is known as the Calcium Carbonate Equivalent (CCE) and is described as a percentage of $CaCO_3$ or its equivalent in the liming material (Zhang

et al., 2014). The largest determining factor of the fineness needed is determined by the CEC of the soil (Haby and Leonard, 2002). For example, in lower CEC (acidic) soils, a lime with a smaller surface area is more desirable because it will have a slower release into the soil and the effects of the lime may last longer on the overall soil pH (Haby and Leonard, 2002). The combination of the active ingredient (CCE) concentration and surface area (fineness) are key components in the selection of an appropriate liming source and are known as the Effective Calcium Carbonate Equivalent (ECCE) of the liming material (Haby and Leonard, 2002; Havlin et al., 2013; Zhang et al., 2014). Liming an acidic soil depends largely on the clay content and OM of the soil; also known as the soil's buffer capacity. Soils with a higher clay content and OM require more lime to raise pH than those with a lower buffer capacity (Havlin et al., 2013).

Table 2 Different liming materials, their chemical composition, and percent calcium carbonate equivalent (Modified from Havlin et al., 2013 and Jones, 2012).

Liming Material	**Chemical Composition**	**CCE %**
Calcium carbonate	$CaCO_3$ (pure)	100
Calcium limestone (aglime)	$CaCO_3$	80-100
Suspension or fluid lime	$CaCO_3$	95-100
Dolomitic limestone	$CaMg(CO_3)_2$	95-100
Dolomite	$CaMg(CO_3)_2$	100-120
Marl (Selma chalk)	$CaCO_3$	70-90
Burned lime	CaO	150-175
Calcium hydroxide (Hydrated or slaked lime)	$Ca(OH)_2$	120-135
Calcium silicate	$CaSiO_3$	80-90
Slag	CaO	60-90
Wood ash	Ca, Mg, K oxides	30-70
Power plant ash	Ca, Mg, K oxides	25-50
Ground oyster shells	$CaCO_3$	Up to 95
Cement kiln dusts	Ca oxides	40-100
Biosolids and by-products	CaO, $Ca(OH)_2$	Variable

Ultimately, the addition of liming materials on an acidic soil raises the pH and decreases the exchangeable Al^{3+} in the soil solution, reducing risk of toxicity (Havlin et al., 2013).

Table 3 The approximate amount of finely ground lime needed to raise the pH of 7-inch layer of soil (Modified from Jones, 2012)

	Lime Requirement (Mg ha^{-1})	
Soil Texture	**From pH 4.5 to 5.8**	**From pH 5.5 to 6.5**
Sand and loamy sand	1.11	1.35
Sandy loam	1.78	2.88
Loam	2.64	3.75
Silt loam	3.32	4.42
Clay loam	4.18	5.09
Clay	8.36	5.14

Alternative Liming Sources

As an alternative to liming, adding P fertilizer to acidic soils has been shown to reduce Al^{3+} toxicity around plant roots (Lollato et al., 2013). Banding phosphorus can be helpful at the beginning of a growing season at planting and can help short term as the plant begins growing to tolerate low pH better at emergence and critical growing stages (Lollato et al., 2013). However, since P is immobile in the soil, banding P fertilizer creates temporary acidic pockets at the site of application after dissolving, but have a short term effect on soil pH as a whole (Havlin et al., 2015).

Biochar

Biochar is a solid organic material created by burning organic materials in an oxygen-limited environment through thermochemical conversion (IBI, 2018; Sandhu et al., 2017). This process is known as pyrolysis or charring and is done at temperatures above 250˚C (Lehmann and Joseph, 2015). This results in a potential fertilizer and liming source that could also increase C sequestration in the soil and improve natural soil fertility dynamics (Sandhu et al., 2017). Biochar and other organic materials have been used as fertilizer in many production systems around the world for many years, however, their effect on soil chemical properties has sparked more interest into their potential as a soil amendment (Mierzwa-Hersztek et al., 2016).

Biochar made from numerous different organic materials such as plant biomass, animal wastes, and sewage sludge have been used as soil additions to aid in many soil chemical processes (Mierzwa-Hersztek et al., 2016). Wang and Liu, (2017) found that animal waste, specifically yak manure, resulted in higher yields of biochar than many other plant materials. However, plant derived (cottonseed hull) biochar was more effective in the removal of heavy metals from soil (Wang and Liu, 2017). Biochar derived from manure has been found to influence and increase the accessibility of plants to essential nutrients than biochar rendered from plants (Brantley et al., 2016). Biochar has been known to absorb nutrients in the soil and can either render them immobile or increase their availability to the plant (Brantley et al., 2016). This can be helpful when attempting to alleviate a toxic nutrient from the soil to allow the plant to thrive in an otherwise harmful soil. Normally, this has been done by adding and incorporating biochar into the soil and then activating by moisture from rainfall.

The effects of biochar addition on soil can vary greatly depending on the type of biochar, application rate, and pyrolysis technique (Mierzwa-Hersztek et al., 2016; Lehman and Joseph, 2015). Biochar pH are generally alkaline and can increase as pyrolysis temperatures increase (Mierzwa-Hersztek et al., 2016). Because it's neutralizing properties, both increasing and decreasing soil pH, biochar is continuously compared to agriculture lime (Lehmann and Joseph, 2015; Yuan and Xu, 2010). Also similar to most liming sources, biochar is added to the soil on a tonnage basis.

CHAPTER III

METHODOLOGY

Research trials were established during the 2015 growing season at EFAW in Stillwater, OK (EFAW) and the South Central Research Station in Chickasha, OK (Chickasha). Additional locations were established in 2016 at North 40 in Stillwater, Oklahoma (N40) and the Cimarron Research Station in Perkins, Oklahoma (Perkins). Trials could not be located in the same locations between years as a means to minimize in-season disease and insect pressure. Treatments were arranged in a split plot design with soil pH as the main plot and winter canola cultivar as the sub-plot. Soil pH treatments were targeted soil pH values. Each location had a different pH gradient (Table 5). pH gradients were pre-existing at Chickasha, EFAW, and Perkins. EFAW and Chickasha pH gradients were established during the 2012-2013 growing season (Lollato and Edwards, 2015). Perkins pH gradients were established during the 2009-2010 growing season (Butchee et al., 2012; Sutradhar et al., 2014). pH gradients at N40 were established ahead of planting in 2016. At all locations, hydrated lime ($Ca(OH)_2$) was used to increase the actual soil pH to the target pH and ammonium sulfate ($Al_2(SO_4)_3$) was used to lower the actual soil pH to the target pH as detailed by Butchee et al. (2012) (Lollato and Edwards, 2015; Sutradhar et al., 2014).

Table 4 Coordinates, soil series, and soil taxonomic classes for trial locations.

Location	Coordinates	Soil Series	Taxonomic Class
Chickasha	35.045953, -97.910845	Dale silt loam	Fine-silty, mixed, superactive, thermic Pachic Haplustolls
		McLain silty clay loam	Fine, mixed, superactive, thermic Pachic Argiustolls
EFAW	36.134768, -97.013399	Easpur loam	Fine-loamy, mixed, superactive, thermic Fluventic Haplustolls
N40	36.137125, -97.079503	Renfrow loam	Fine, mixed, superactive, thermic Udertic Paleustolls
		Kirkland silt loam	Fine, mixed, superactive, thermic Udertic Paleustolls
Perkins	35.999280, -97.039092	Dougherty loamy fine sand	Loamy, mixed, active, thermic Arenic Haplustalfs
		Konawa fine sandy loam	Fine-loamy, mixed, active, thermic Ultic Haplustalfs

Table 5 pH gradients for each location after adjustment and trials.

Location	pH Gradient		
	Low	Median	High
Chickasha	3.88	4.97	6.06
EFAW	3.63	5.24	6.85
N40	3.84	5.31	6.78
Perkins	3.18	4.46	5.73

Sub-plots consisted of four commonly grown winter canola cultivars, including: DKW 41-10, 44-10, 45-25, and 46-15. These cultivars were selected not only to provide additional information on commonly grown cultivars in the southern Great Plains but to build on the existing data in winter canola production as previous work had only evaluated non-glyphosate tolerant cultivars (Lofton et al., 2010). For both main and sub-plots, treatments were arranged in a randomized complete block design. Plot dimensions were 6.67 meters wide and 6.67 meters long for each main plot. These were further divided into four subplots with 1.67 meter width, while maintaining the same length. In 2015-16 locations in EFAW and Chickasha, treatments were replicated four times. However, due to space limitations, treatments were only replicated three times in Perkins and N40 in 2016-17. Composite soil samples were collected prior to

establishment each year to determine soil N, P, and K concentrations to aid in soil fertility applications.

All plots for both years were planted using a 6200 Monosem vacuum planter (Monosem Inc., Edwardsville, KS). Plots were planted at the rate of 790,000 seeds ha^{-1}. All plots were planted on 38 cm spacing. All weeds, insects, and diseases were controlled using commercially available pesticides as needed based on current winter canola recommendations through Oklahoma State University.

Winter canola growth measurements were collected for all locations in 2015-16 and 2016-17. Final emergence and winter survival ratings were collected in-season and final yields were determined at maturity. Plant counts were taken from the middle two rows of each treatment four weeks following planting to determine final emergence values. Additional plant stands were collected at spring green-up. The difference between final emergence and spring stand counts were used to determine winter survival. At maturity, all plots were swathed, windrowed, and allowed to dry-down for seven days. Following dry-down, plots were mechanically harvested using a small-plot combine with a custom built pick-up attachment to the grain header. Plot weights were used to determine yield on a per hectare basis and adjusted to 10% moisture concentrations. From each plot, subsamples were collected. These subsamples were used to analyze oil and protein concentration with a Near-InfraRed Reflectance (NIR) spectrometer (Perten Instruments, Hägersten, Sweden).

Field Study Soil Analysis

Following harvest, soils were collected from every subplot at all locations and replications. A minimum of 15 soils to the depth of 15cm were collected from each plot and homogenized. Samples were placed in drying overs, dried at 67°C for 72 hours, and ground to pass a 2-mm sieve. Soil pH was determined using a 1:1 soil:water ratio (10g of soil to 10mL of deionized water) and measured using a glass electron probe (Mettler-Toledo, LLC, Columbus,

OH). Soil Al^{3+} was extracted using a 1:10 soil to 1M KCl solution, where soil and solution were mixed in 100 mL plastic cups, shaken using an orbital shaker for 30 minutes, and extracts filtered using a Q2 quantitative filter paper. Extracts were analyzed using a coupled plasma atomic emission spectroscopy (ICP-AES) (Spectro Arcos, Kleve, Germany).

Field Study Statistical Analysis

Data were analyzed with SAS version 9.4. Regression analysis was conducted between soil pH and soil aluminum concentration to canola grain yields using Procedure Reg. Procedure NLIN was used to create linear plateaus to show the critical pH level at which yield was no longer affected by soil pH. Locations and/or varieties that did not fit a linear plateau were fit to a linear regression line using PROC REG, all at $\alpha = 0.05$. Location, cultivar, pH, and Al^{3+} were analyzed separately. Comparisons were made between pH and grain yield, Al^{3+} and grain yield, and pH and Al^{3+}, for each cultivar at each location.

Greenhouse Study

In addition to field studies, a greenhouse study was established in late spring of 2017 to determine the ability of utilizing biochar to alleviate Al^{3+} concentrations in the soil. Greenhouse trials were arranged in a randomized complete block design with biochar type as the main plot and a complete factorial of biochar rate and Al^{3+} concentration in the subplot. Finally, three different biochar rates were evaluated: 2.24 Mg ha^{-1} (1 T $acre^{-1}$), 5.6 Mg ha^{-1} (2.5 T $acre^{-1}$), and 11.21 Mg ha^{-1} (5 T $acre^{-1}$) as well as a replicated check with no added biochar. The biochar type that was used was from poultry litter. Soils for the greenhouse trial were collected from the North Central Research Station location in Lahoma and Al^{3+}concentration evaluated for each. Biochar was incorporated into the soil by hand and scheduled watering dates and times mimicked rainfall events as closely as possible and aided in biochar activation. The greenhouse study took place over a three-week period.

Greenhouse Soil Analysis

At the end of the greenhouse trial, subsamples were taken from each pot for analysis. Samples were placed in drying overs, dried at 67°C for 72 hours, and ground to pass a 2-mm sieve. Soil Al^{3+} was determined in the same manner as described for the field study.

Greenhouse Statistical Analysis

Data were analyzed with SAS version 9.4. Analysis of variance was conducted through Procedure Mixed. In this analysis, biochar application rate was considered a fixed effect while replications was considered random. An LSD means separation test was conducted with a Tukey modifier to determine significant differences between treatment means, at $\alpha = 0.05$.

CHAPTER IV

RESULTS AND DISCUSSION

Weather

Ideal conditions at planting greatly influenced the EFAW site and helped the crop to establish itself well in order to thrive in the upcoming growing season. Above average temperatures and frequent precipitation events around planting and seedling development helped in root development and elongation and allowed the crop to establish a root system well into the soil profile. This aided in the plant to establish enough vegetative growth in order to avoid low temperatures and winter kill early in the season.

Conditions at planting for N40 in the 2016 growing season were not as ideal as the previous season at EFAW although they are close in proximity. While there were rain events and more moisture present, they were very low and infrequent and paired with higher temperatures that could affect the success of the plant from a very early stage.

Rain events for Perkins were more infrequent and collected precipitation was low at each event while temperatures generally remained high. Due to the sandy soil properties at Perkins, moisture was not able to accumulate and be conserved in the soil profile as well. This

caused issues in the establishment and success of the crop due to the low and infrequent precipitation and the inability of the soil to provide sufficient water holding capabilities of the little amount of water applied.

Precipitation was ideal but temperatures were high at planting for Chickasha. While there was adequate moisture for germination and seedling growth, persistent warm temperatures could be a contributor to excessive growth before winter and can cause lower winter survivability. Although precipitation was ideal at planting, it was lower following planting. However, the silty soil could contribute to the moisture conservation for use by the plant.

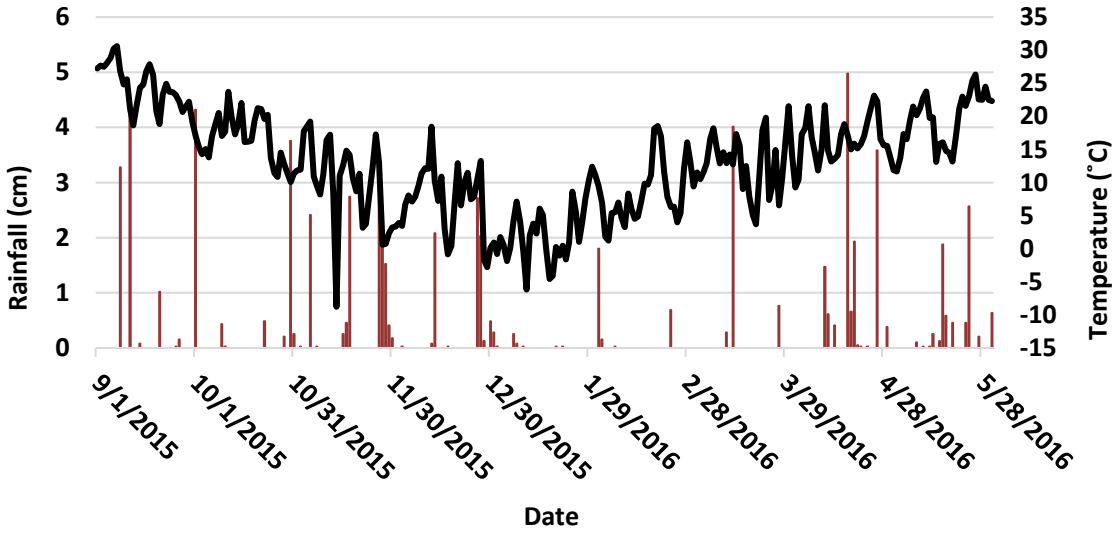

Figure 3 Rainfall and Temperature for Stillwater (EFAW) for the 2015-2016 growing season.

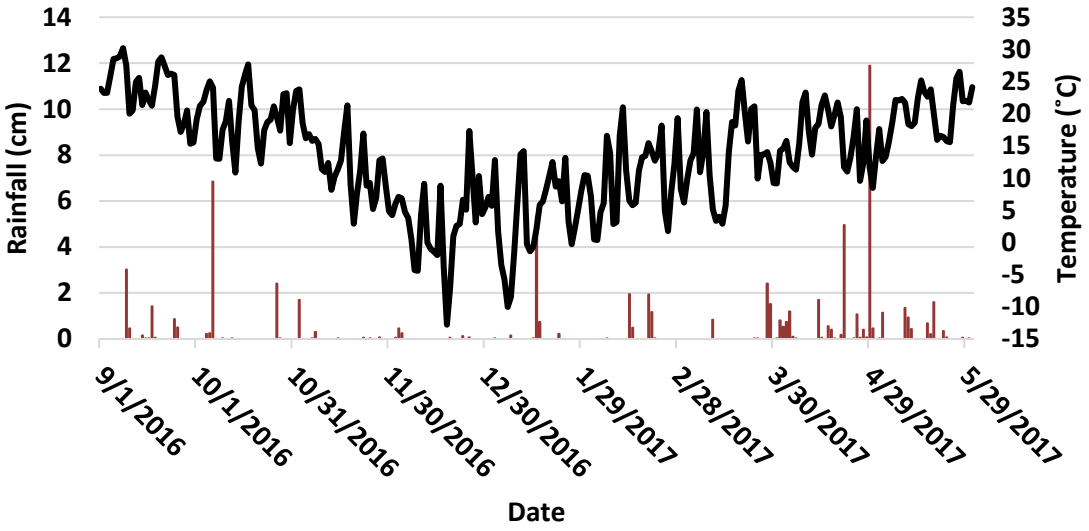

Figure 4 Rainfall and Temperature for Stillwater (N40) for the 2016-2017 growing season.

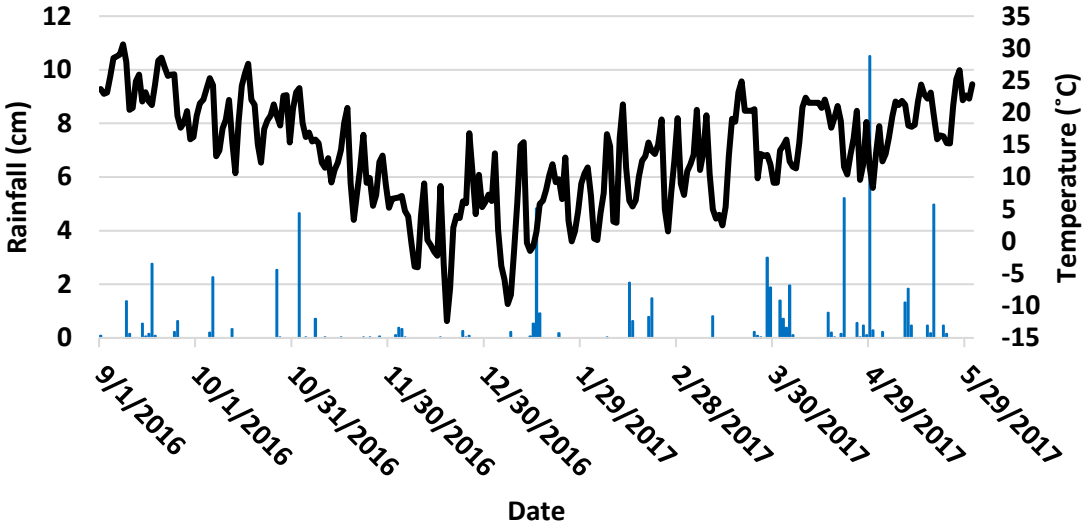

Figure 5 Rainfall and Temperature for Perkins for the 2016-2017 growing season.

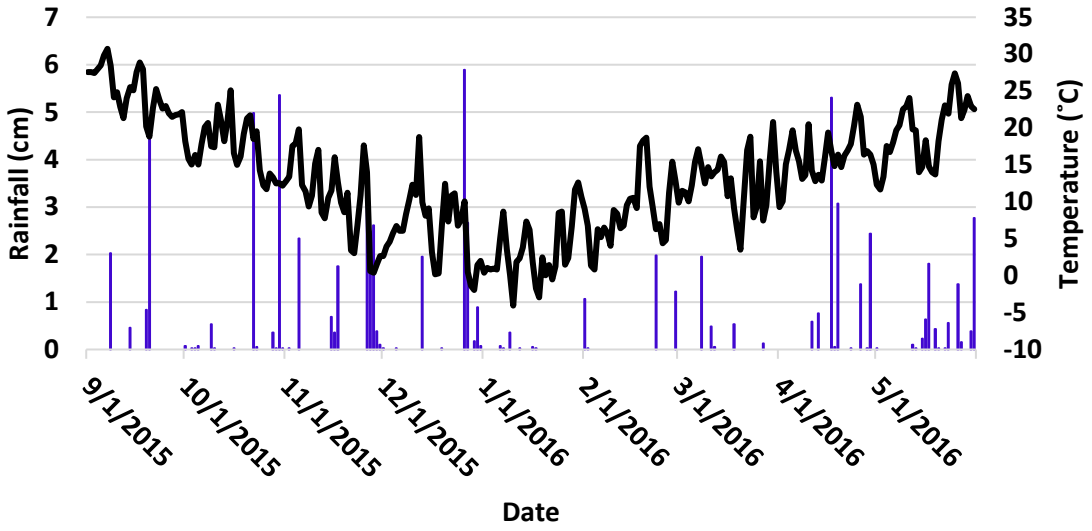

Figure 6 Rainfall and Temperature for Chickasha for the 2015-2016 growing season.

Crop Yield

Canola grain yields ranged greatly across both years and all locations evaluated in this project. Highest and lowest yields were found at the EFAW location in Stillwater, Oklahoma during the 2015-2016 season. Yields at this location ranged from 14 through 3037 kg ha^{-1} with an average of 1939 kg ha^{-1}. Interestingly, no zero yielding plots were apparent. This opposes previous literature that suggested a critical soil pH where canola cannot grow (Lofton et al., 2010). While this could indicate better establishment and growing conditions, it also could indicate lower susceptibility of the evaluated cultivars to soil acidity.

Soil pH and Yield

Overall, there was a general positive trend between soil pH and winter canola yields, indicating that as soil pH was increased, canola yields increased similarly (Figure 7). However, there is some variability in the model. This variability not only encompasses the variability in yield potential at the individual locations but also the individual locations evaluated appear to be separate. This indicated that the response of winter canola to soil pH differed by location.

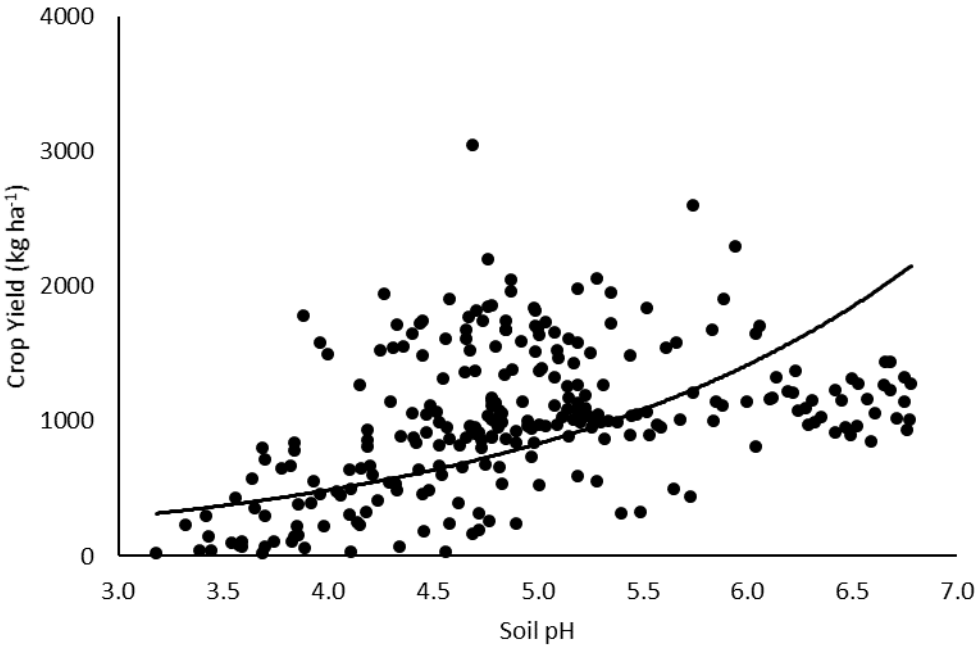

Figure 7 Relationship between soil pH and winter canola yield.

When evaluating the response of soil pH and canola yields individually between locations, a strong linear relationship was noted at the N40 location (r^2 = 0.59; p-value = <0.001), with a 12.2 kg ha^{-1} increase in yields associated with a 0.1 increase in soil pH. A significant linear relationship was also found at the EFAW location in 2016 but the relationship was weaker (r^2 = 0.21; p-value = 0.035). However, there was a greater response to increasing pH with a 46.7 kg ha^{-1} increase in canola grain yield at 0.1 increase in soil pH. Figure 7 highlights the relationship between soil pH and winter canola yield at Chickasha, N40, and Perkins locations when averaged across canola cultivars. EFAW was omitted from figure 7 because it did not follow the same trend as the other locations; pH and crop yield relationships at EFAW were fit to linear plateau models. No significant relationship was found between soil pH and crop yield for both the Chickasha and Perkins locations. However, it should be noted that the significance value at the Perkins location was close to significant (p-value = 0.0547), where if alpha values were increased to 0.10 a significant relationship could have been measured.

Table 7 Relationships between soil pH and yield, averaged across locations.

Year	Site	Equation	Adjusted r^2	p-value
2016	Chickasha	159x+714	0.03	0.297
2016	EFAW	467x-162	0.21	0.035
2017	N40	122x+352	0.59	<0.001
2017	Perkins	139x-245	0.17	0.0547

Impact of Soil pH on Individual Cultivars

While there has not been a dedicated effort to breed soil acidity tolerance into current winter canola germplasm, differences in winter canola cultivars have been documented. Table 8 lists each Dekalb winter canola variety with its respective color displayed in each of the following figures and tables for ease of organization and identification. These colors will be used to consistently refer to the individual cultivars throughout the remainder of the document. Table 9 documents the response of the tested winter canola cultivars on soil pH.

Table 8 Dekalb winter canola cultivars analyzed, listed with their corresponding color for identification in following figures and tables.

Cultivar	Color
41-10	Blue
44-10	Orange
45-25	Green
46-15	Yellow

Table 9 Relationships between soil pH and yield across all locations in 2015-16 and 2016-17 season. (NS= Not Significant; S= Significant)

Year	Site	Cultivar	Equation	Joint	Adjusted r^2	P-value
2016	Chickasha	41-10	213 x+311	NS	-0.01	0.38 NS
		44-10	243x+266	NS	0.13	0.09 NS
		45-25	321x+17	NS	0.11	0.07 NS
		46-15	60x+1278	NS	-0.04	0.78 NS
2016	EFAW	41-10	2978x-10271	4.15		0.02 S
		44-10	5533x-20560	4.07		<0.01 S
		45-25	9011x-32601	NS		0.11 NS
		46-15	4203x-14653	4.10		0.04 S
2017	N40	41-10	41x+772	NS	0.30	0.17 NS
		44-10	133x+312	NS	0.78	<0.01 S
		45-25	151x+232	NS	0.58	<0.01 S
		46-15	100x+450	NS	0.62	<0.01 S
2017	Perkins	41-10	158x-372	NS	0.30	0.02 S
		44-10	99x-7	NS	0.01	0.30 NS
		45-25	129x-58	NS	0.01	0.29 NS
		46-15	164x-346	NS	0.29	0.01 S

Both EFAW and N40 locations were found to have a significant relationship when averaged across cultivar, and fairly consistent positive relationships were found between yield and soil pH among cultivars. In fact, significant linear plateaus could be found at the EFAW locations for three of the four cultivars evaluated. Critical values for these cultivars were found to range from 3.9 through 4.2. Beyond these soil pH values, winter canola yields did not significantly vary with continually increasing soil pH. These critical values are much lower than that previously documented by Lofton et al. (2010), which was found to be around 5.8. This was due to the amount of useful data points after the joint that illustrated a clear plateau in yield after the critical pH value was reached. No other significant critical soil pH were found. This means that winter canola yields continued to increase with increasing pH for the specific range tested.

The EFAW site was most likely the only site to exhibit a critical pH value over all sites due to its inclination to success early on in the growing season. EFAW experienced the most ideal environmental conditions at planting and winter canola was able to quickly establish itself in that

site. Since management in this study was only done in the top 10-15cm and canola taproots can quickly reach through those depths early on in its lifecycle, it is possible that the success of the crop. This could allow the plant to grow through the pH management layers quickly and before soil pH is a major defining factor in reproductive development, thereby not allowing for a true critical soil pH to be determined. Prompt plant establishment in the beginning of the growing season also aided in battling winter kill and more plants were able to survive to harvest.

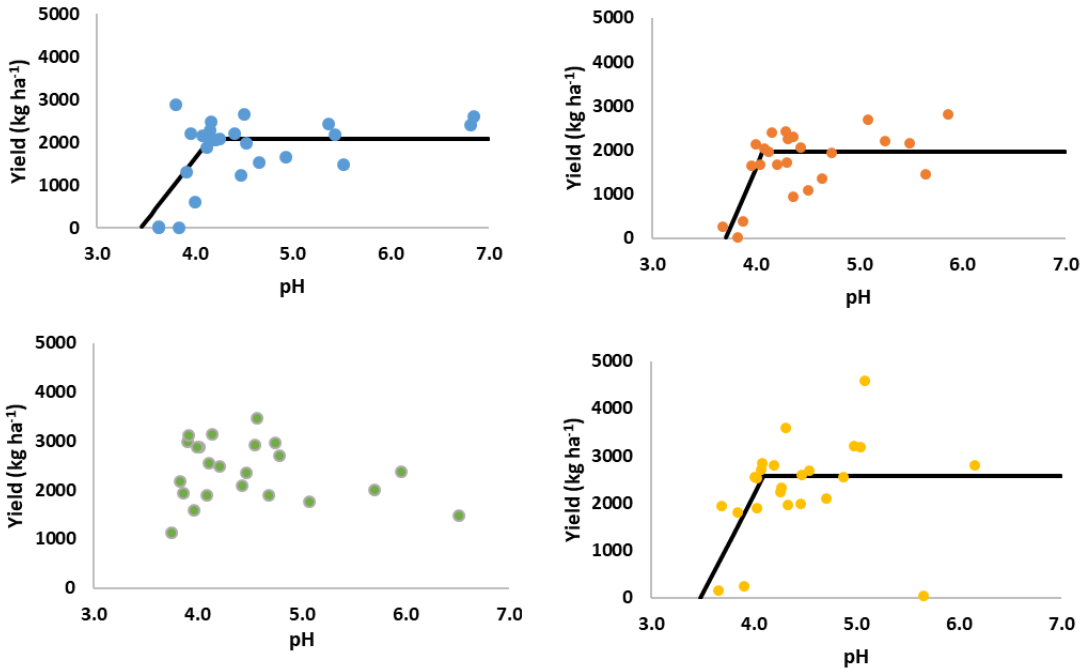

Figure 8 Relationships between soil pH and yield for each cultivar, including plateaus at EFAW

Previously mentioned, a significant relationship between winter canola and soil pH were not found at the Perkins locations but critical values were close. Part of the explanation for this is the differing response of the cultivars evaluated. Figure 9 indicates that DKW 41-10 and 46-15 had a significantly positive relationship between canola yields and soil pH, while both 44-10 and 45-25 did not. These relationships showed that canola yields increased 15.8 and 16.4 kg ha^{-1} for every 0.1 increase in soil pH for DKW 41-10 and 46-15, respectively.

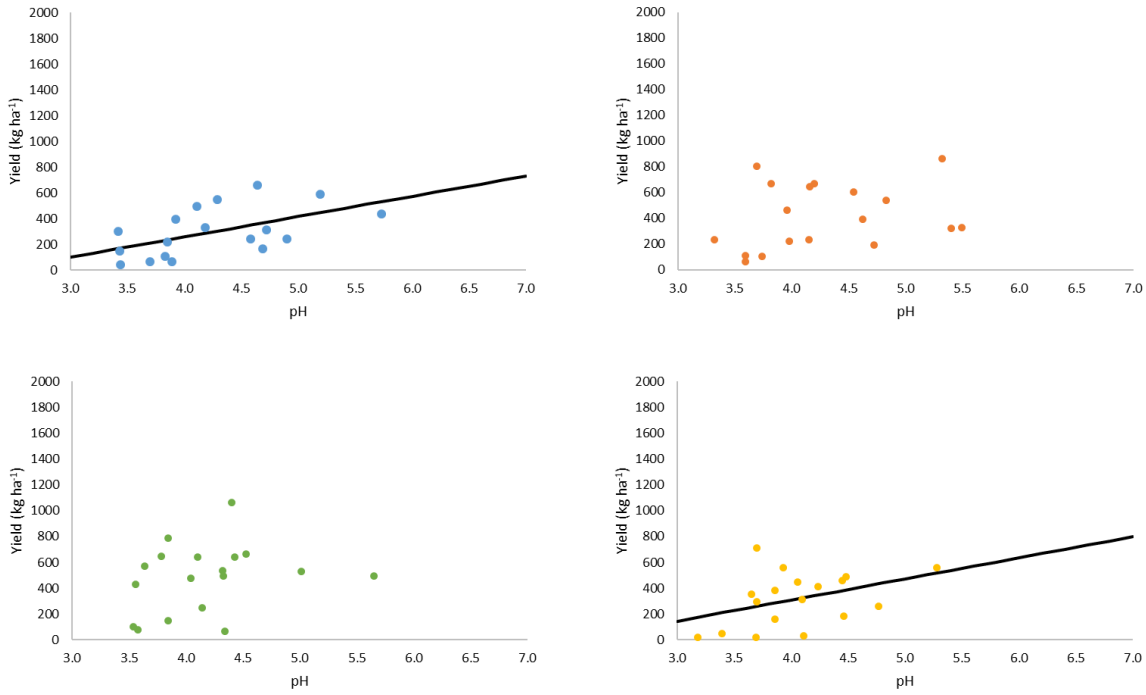

Figure 9 Relationships between soil pH and yield for each cultivar at Perkins

The Chickasha location was not found to have a significant relationship among cultivars individually or when averaged. There was also no significant relationships between canola yields and soil pH for any cultivars evaluated. This could indicate some other factor limiting or influencing yields, which will be discussed in the following sections.

Soil pH and Al

Generally, soil pH and extractable Al^{3+}concentration were negatively related, fitting a non-linear relationship (r^2=0.27). However, multiple distinct groupings within the model can be noted. Figure 10 shows the relationship between soil pH and extractable Al^{3+} at the different site years. When evaluated independently, all locations had a strong significant inverse relationship between soil pH and extractable aluminum concentration. The difference in site years could be explained by the temporal and spatial variability of the sites. Not only are sites different, resulting in varied soils and soil classification, differences in sampling conditions at time of collection

could also alter the relationship between soil pH and extractable Al^{3+} as well (Ferguson et al., 2007).

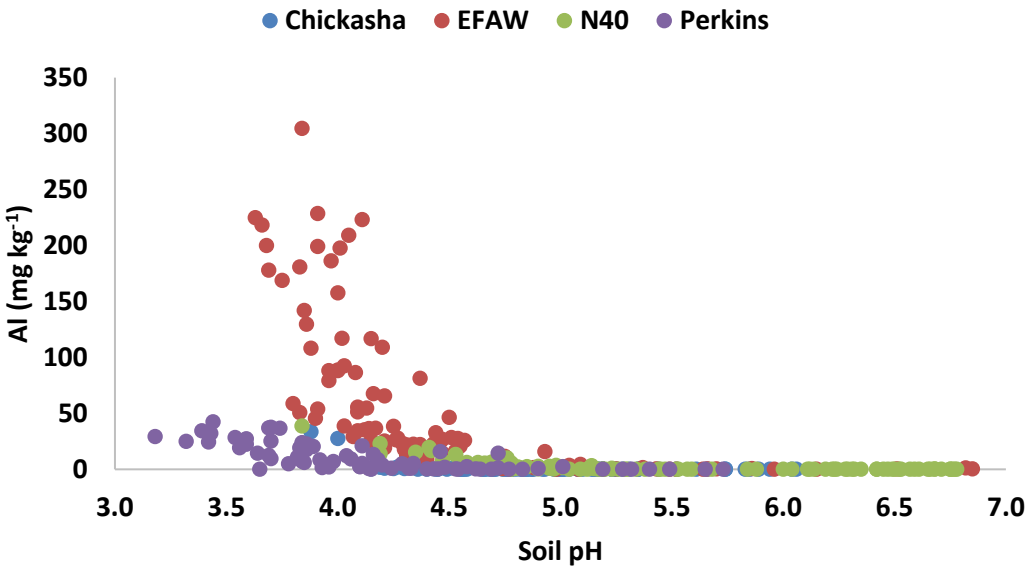

Figure 10 Relationship between soil pH and extractable Al^{3+} at each location

Al and Crop Yield

When averaged across site-years, crop yields were not significantly related to extractable Al^{3+} concentrations. This is due to the high amount of variability noted in the model. This should be expected as both soil pH and crop yield as well as soil pH and extractable Al^{3+} concentrations varied between locations analyzed.

When analyzed separately, crop yield was negatively related to extractable Al^{3+} content in all locations, with the exception of Chickasha. Both N40 and Perkins location fit best into a linear relationship, with a relationship of 0.29 and 0.47 for N40 and Perkins, respectively. The EFAW location best fit into a non-linear regression (r^2= 0.43) with a limited decrease in yield at low level Al^{3+} levels with a greater decrease following 50 mg kg^{-1} of Al^{3+}. While a critical pH value cannot be accurately determined, a notable relationship between pH and yield, Al^{3+} and yield, and pH and Al^{3+} can be seen in all locations except Chickasha. Explanations for the lack of

yield at Chickasha could include different environmental conditions or other soil chemical properties. Results suggest soil Al^{3+} is naturally low at Chickasha because the change in pH did not affect the Al^{3+} availability in solution. Lofton et al. (2010) found a critical Al^{3+} concentration between 11.3 to 14.7 mg kg^{-1} at which canola yield began to decline. Since many yield responses were variable below the previously documented critical Al^{3+} concentration, regardless of pH, it is assumed that other factors are influencing yield not associated with soil acidity or Al^{3+} concentration.

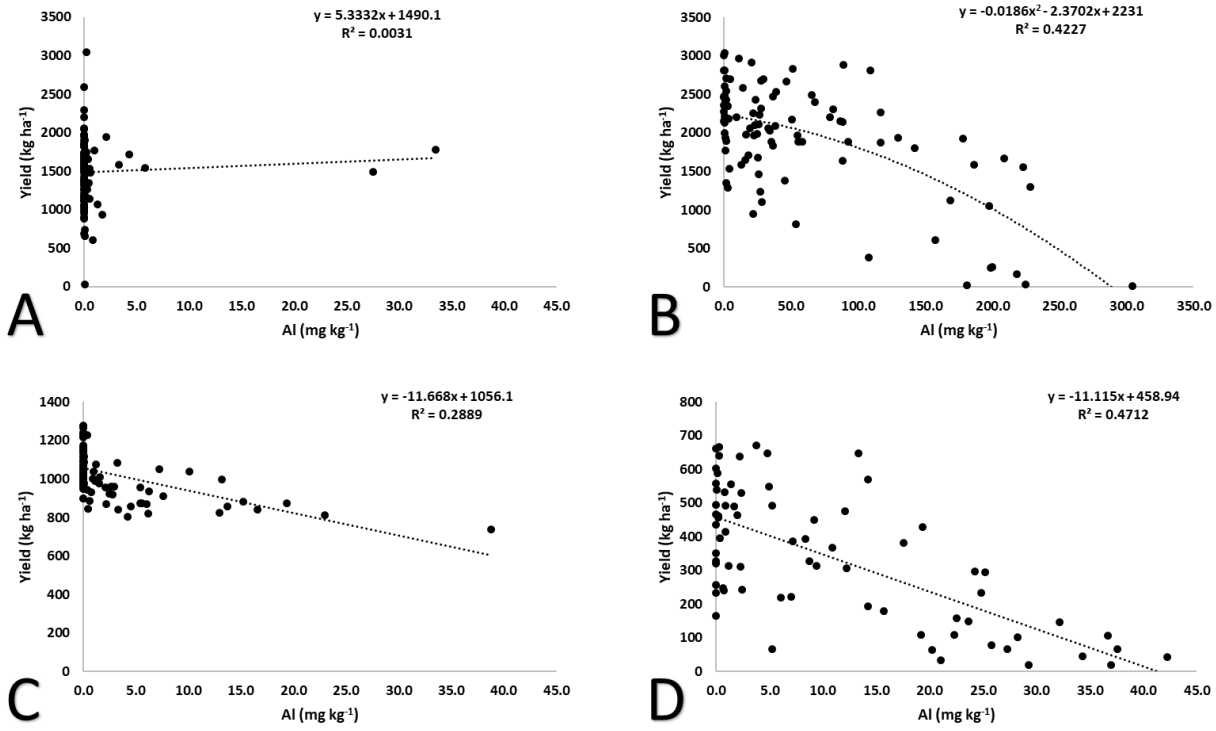

Figure 11 Al and crop yield for each location. Chickasha: A, EFAW: B, N40: C, and Perkins: D.

Greenhouse Results

Analysis of poultry litter biochar was done after pyrolysis and before application to the soil in the greenhouse trial. Three samples were taken and analyzed from the batch and measurements were averaged. Table 10 lists the factors analyzed and the average of the batch.

Table 10 Analysis of poultry litter biochar used in greenhouse trial.

Factor	Average	Units of Measure
pH	10.3	-
P	3.49	%
Ca	10.56	%
K	5.83	%
Mg	2.29	%
Na	2.67	%
S	1.42	%
Fe	32822.57	mg kg^{-1}
Zn	1817.07	mg kg^{-1}
Cu	295.64	mg kg^{-1}
Mn	7874.24	mg kg^{-1}
B	138.30	mg kg^{-1}
Ni	38.43	mg kg^{-1}
Mo	29.67	mg kg^{-1}
Co	13.41	mg kg^{-1}
Se	32.77	mg kg^{-1}
W	52.82	mg kg^{-1}
TN	1.50	%
TC	19.56	%

Applications of biochar greatly decreased the KCl-extractable Al^{3+} concentration in the soil (Figure 11). A significant decrease in extractable Al^{3+} concentrations was found with the application of the lowest rate of biochar, 2.24 Mg ha^{-1} (1 T ac^{-1}). A difference of 28.23 mg kg^{-1} was observed from 0 Mg ha^{-1} to 2.24 Mg ha^{-1} (p-value of 0.0038). In the following applications, the decrease in Al^{3+} concentration was not significant, although concentration continued to decrease as more biochar was applied. While the application of 2.24 Mg ha^{-1} (1 T ac^{-1}) did result in significant declines in extractable Al^{3+}, following the application of 5.56 Mg ha^{-1} (2 T ac^{-1}), virtually no extractable Al^{3+} was noted.

These results can be compared to other research where biochar was used as a liming source in order to render toxic elements unavailable as well as those where biochar was used as a means of alleviation. Hass et al. (2012) found that although biochar increased pH and caused

other essential plant micronutrients like Mg, Cu, and Zn to be more available, it also decreased the availability of some plant essential macronutrients that depend on lower soil pH, such as P, K, and S. In addition to altering the availability of elements in the soil solution, other research has documented the toxic element alleviation capabilities of biochar. Lin et al. (2018) documented the alleviation of Al^{3+} from the soil solution by analyzing the Al^{3+} uptake by cabbage plants. Al^{3+} content in the cabbage plants fell at least 12% from each of the three biochar treatment types (Lin et al., 2018). Therefore, while biochar can hinder other plant metabolic processes due to the decreased availability of those macronutrients caused by biochar application, it can also be beneficial in alleviating toxic elements from the soil as well.

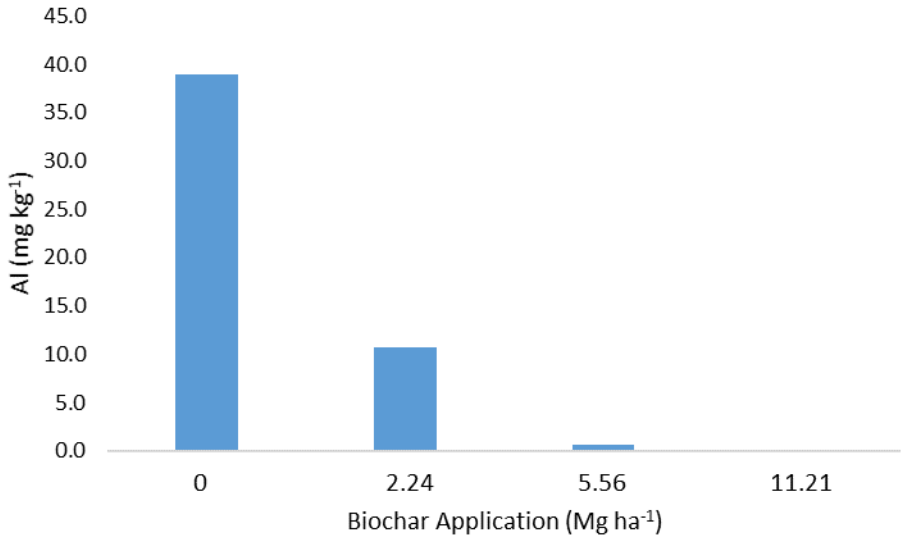

Figure 12 Biochar application replication averages with statistical significances.

CHAPTER V

CONCLUSION

Field Study

Overall, pH is very influential to winter canola yield. Although critical soil pH levels were not found for a majority of the locations evaluated, this does not mean one does not exist. In a majority of locations, fewer data points than desired were observed above 6.0 soil pH. This might not allow for a critical pH to be determined. However, with adequate data points at and below 5.8, there is an indication that critical pH would be above these values.

In soils where available/exchangeable Al^{3+} concentration exists naturally in the soil system, both soil pH and exchangeable Al^{3+} influenced canola grain yields. In these systems, soil pH and Al^{3+} were well related and soil pH was a good indicator of canola yield potential. Where Al^{3+} concentrations were low and the relationship between Al^{3+} and soil pH were not significant, canola yields were not directly related to soil pH. When soils are naturally low in Al^{3+}, there is little to no yield response associated with low pH affecting present concentration. This resulted in fluctuating soil pH but only minor changes in canola grain yields. This would indicate that, in these conditions, soil pH analysis would have little indication on canola grain potential. Pairing

soil pH analysis with an evaluation of exchangeable Al^{3+} would provide a better indication on the impact low soil pH would have on canola grain yields. Where Al^{3+} concentration is naturally low in the soil solution, indifferent of soil pH, yield response is due to other environmental factors. While there were no other samples taken or tests run to determine other factors influencing yield loss, it can be speculated that other elemental toxicities or environmental conditions had an influence on observed results. In specific cases like Chickasha where responses were scattered indifferent of the soil pH and Al^{3+} concentration, weather and other soil factors could be considered to provide further explanation of observed outcome.

Greenhouse Study

Data suggests that biochar is an effective source of Al^{3+} alleviation from the soil. Overall, as biochar applications increased, Al^{3+} concentration decreased. While continued application of biochar decreased Al^{3+} concentration in the soil, no significant difference was made after 2.24 Mg ha^{-1} (1 T ac^{-1}). However, following initial applications, low concentrations of Al^{3+} remained in the soil. Higher initial concentrations of Al^{3+} in the soil could result in further alleviation at the higher application rates. It should be noted these higher applications should not be harmful to the soil system but would have little impact on the goal of alleviating Al^{3+} in the soil system.

When converting organic matter into biochar and applying it from a waste management standpoint, biochar will act as a binding agent to alleviate Al^{3+} as well as an organic matter addition to the soil without harm to the soil. This is helpful in waste management because the conversion from waste to biochar results in a much smaller volume of material compared to the raw organic material used in the conversion process. Therefore, excessive amounts of organic material can be converted into less than half the original volume and can be land applied as means of disposal.

After an examination of the differences in biochar types and comparison to lime applications on soil properties, further research would be beneficial to provide more data on biochar and its effectiveness.

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APPENDICES

Chickasha											
	N										
W	+	E									
	S										
		Rep 1			Rep 2			Rep 3			Rep4
	41-10	4.0		44-10	5.5		45-25	4.5		45-25	6.0
	44-10			46-15			46-15			46-15	
	45-25			45-25			44-10			44-10	
	46-15			41-10			41-10			41-10	
	41-10	4.5		46-15	4.5		45-25	7.0		45-25	5.5
	44-10			44-10			44-10			46-15	
	45-25			41-10			41-10			41-10	
	46-15			45-25			46-15			44-10	
	41-10	5.0		46-15	7.0		46-15	6.0		45-25	4.5
	44-10			45-25			44-10			46-15	
	45-25			41-10			45-25			44-10	
	46-15			44-10			41-10			41-10	
	41-10	5.5		44-10	5.0		45-25	4.0		44-10	5.5
	44-10			41-10			44-10			45-25	
	45-25			46-15			41-10			41-10	
	46-15			45-25			46-15			46-15	
	41-10	6.0		46-15	6.0		44-10	5.0		46-15	4.0
	44-10			41-10			41-10			44-10	
	45-25			44-10			45-25			41-10	
	46-15			45-25			46-15			45-25	
	41-10	7.0		41-10	4.0		41-10	5.5		46-15	7.0
	44-10			44-10			46-15			41-10	
	45-25			45-25			45-25			44-10	
	46-15			46-15			44-10			45-25	
		Rep 1			Rep 2			Rep 3			Rep 4

Figure 1A Chickasha plot map from 2015-2016 growing season.

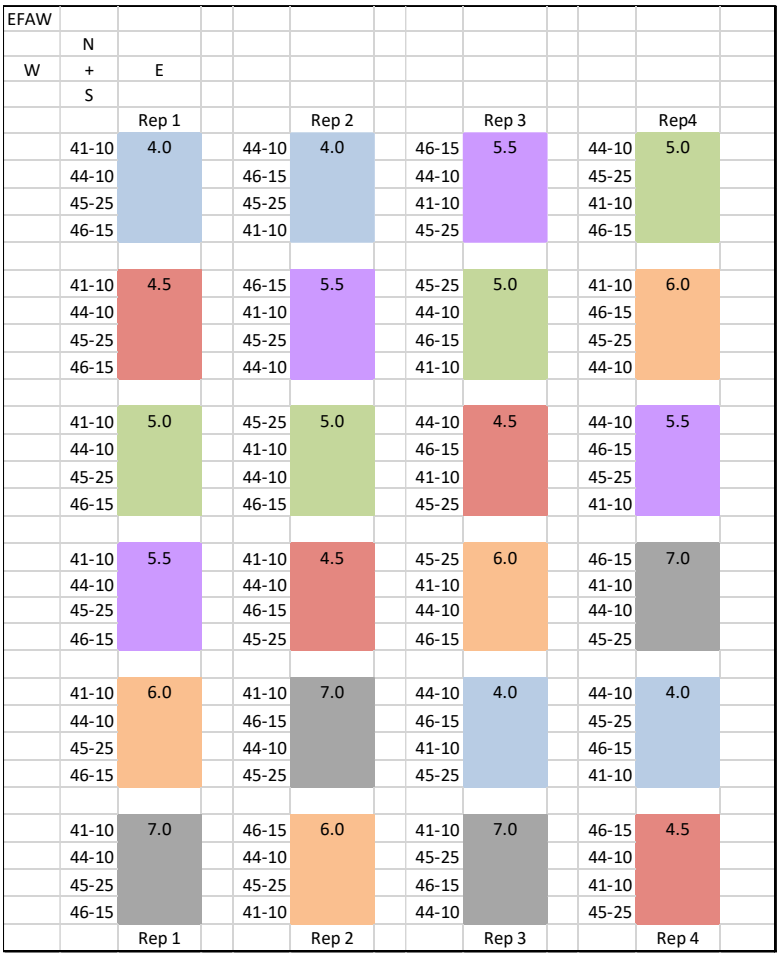

Figure 2A EFAW plot map from 2015-2016 growing season.

Figure 3A North 40 plot map from 2016-2017 growing season.

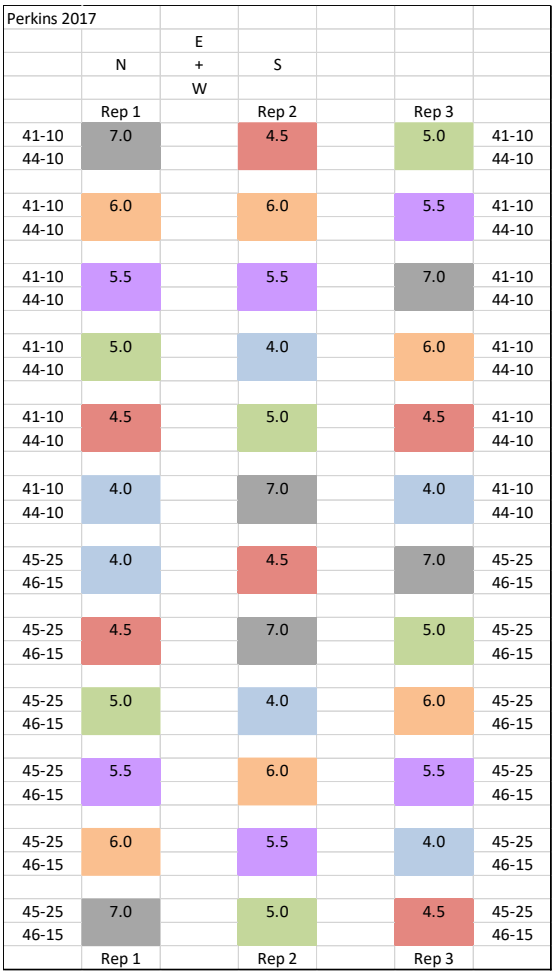

Figure 4A Perkins plot map from 2016-2017 growing season.

Table 1A Crop yield from each location

	Yield (kg ha^{-1})		
Location	**High**	**Average**	**Low**
Chickasha	1977	1401	94
EFAW	3038	1939	15
N40	1279	1025	739
Perkins	672	341	19

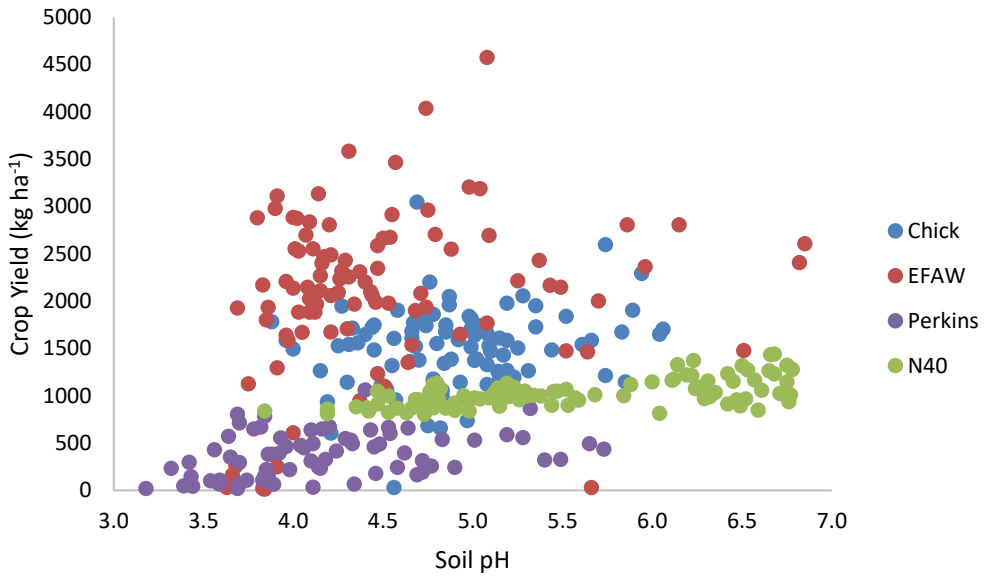

Figure 5A Relationships between soil pH and crop yield at individual locations

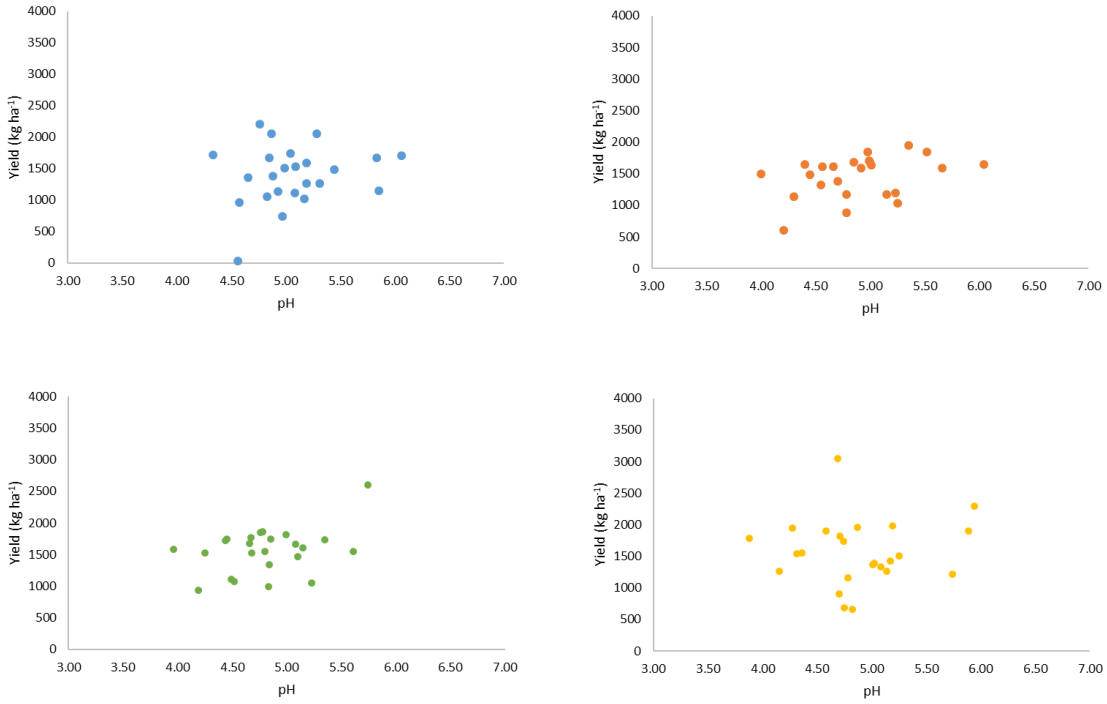

Figure 5A Relationships between soil pH and yield for each cultivar at Chickasha

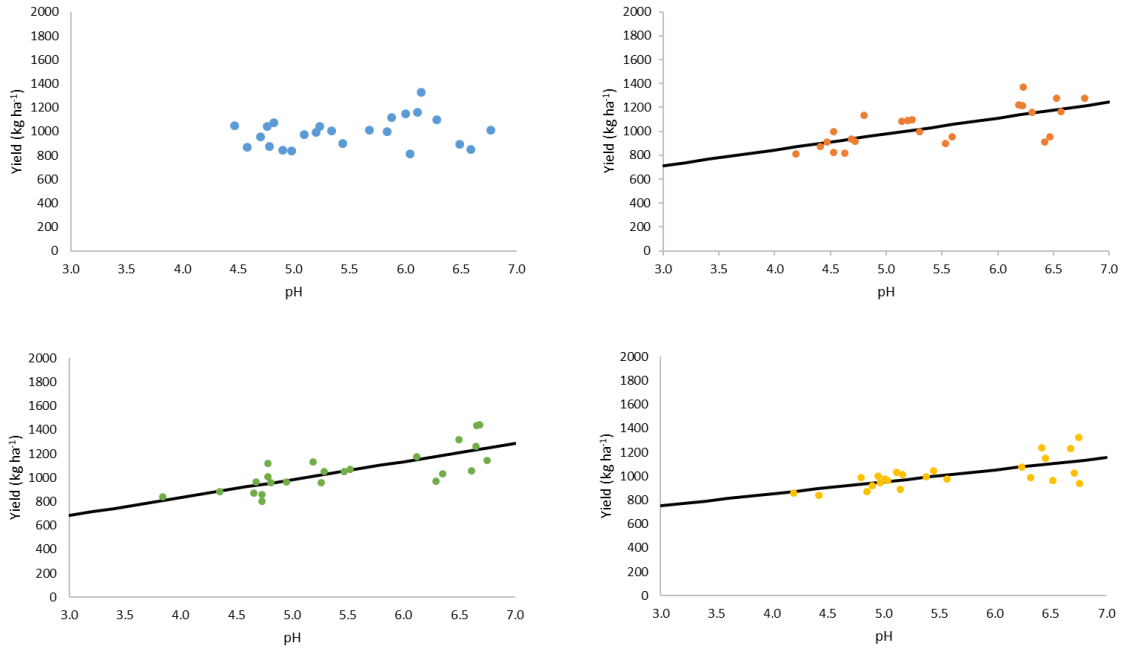

Figure 6A Relationships between soil pH and yield for each cultivar at N40

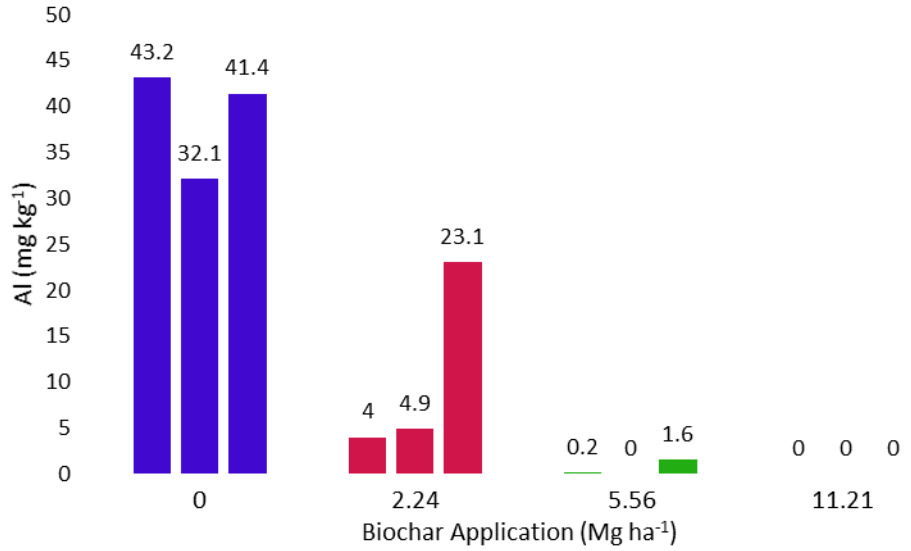

Figure 7A Al^{3+} concentrations for each pot after application

Figure 8A Canola seedlings in each pH gradient block at EFAW

Figure 9A Canola seedlings in each pH gradient block at Perkins

Figure 10A Canola seedlings in each pH gradient block at N40

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